

High Precision Headspace Gas Analysis Using Portable Ball SAW Gas Chromatograph

Takamitsu Iwaya^{1†}, Shingo Akao¹, Tatsuhiko Okano¹, Nobuo Takeda¹, Yusuke Tsukahara¹, Toru Oizumi¹, Hideyuki Fukushi¹, Tomoki Tanaka¹, Maki Sugawara¹, Toshihiro Tsuji^{2,1}, Akinobu Takeda¹, and Kazushi Yamanaka^{1,2}
(¹Ball Wave Inc.; ²Tohoku Univ.)

1. Introduction

Analysis of aroma components at manufacturing and transportation sites is required for product development and quality control of foods and beverages. A gas chromatograph (GC) is useful for the analysis of aroma components, but the large size of conventional GC makes it difficult to apply it to the field. We have developed a portable GC system equipped with a ball surface acoustic wave (SAW) sensor^{1,2)} that the feature is multiple roundtrips of SAW on a spherical crystal: ball SAW GC.^{3,4)} In addition, we have demonstrated its usefulness in the analysis of aroma components in food products.⁵⁾

On the other hand, the sample gas collection method is also important for quantitative analysis with good repeatability. The headspace (HS) method, in which the sample is placed in a vial container and the gas inside the container is injected into the GC, is widely used in the analysis of aroma components. In this study, we investigated the influence of the conditions of HS analysis of *sake* on the measurement results using a ball SAW GC.

2. Headspace method

The HS method is broadly classified into the static HS method and dynamic HS method, as shown in Fig.1. The static HS method collects a fixed concentration of gas phase under equilibrium conditions, thus enabling analysis with good reproducibility. On the other hand, the static HS method has the problem that the equilibrium is disrupted because the inside of the vessel is depressurized when the collected volume increases. In addition, the use of a syringe makes the automated device larger, making it difficult to apply the method to on-site analysis.

Since the dynamic HS method continuously samples the gas phase in a vial container, it can easily collect large volumes of gas and is relatively easy to apply to on-site analysis. On the other hand, there is concern that slow diffusion of volatile components from the sample liquid surface may impair quantitation.

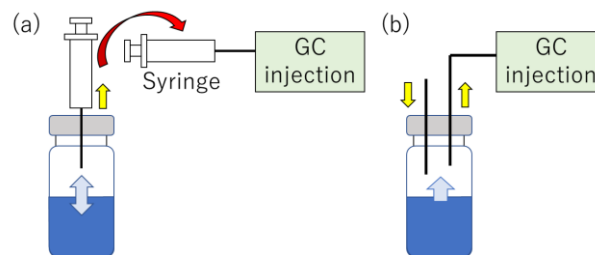


Fig. 1 Schematic view of HS analysis.
(a) Static analysis. (b) Dynamic analysis.

3. Experiment

The analyzer was a portable ball SAW GC (Sylph SY-400), and the aroma components of *sake* were analyzed by the static HS and dynamic HS methods. To collect the same amount of sample gas for both methods, in the static HS method, a gas bag filled with a sufficient amount of liquid was maintained at 20°C and the gas phase portion of the gas bag was aspirated from the collection tube of the ball SAW GC as shown in Fig. 2 in order to eliminate concerns about depressurization.

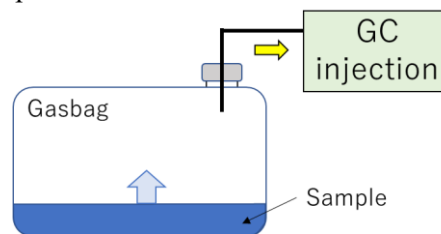


Fig. 2 Static HS analysis using gasbag.

For the dynamic HS method, approximately 10 ml of *sake* was placed in a 22 ml volumetric vial maintained at 20 °C, and the gas phase in the vial was aspirated from the collection tube of a Ball SAW GC.

The ball SAW GC was equipped with a concentrator using Tenax TA as sorbent and a WAX column with an inner diameter of 0.25 mm and a length of 30 m. The ball SAW sensor used a 3.3 mm diameter quartz with a center frequency of 150 MHz coated with PDMS as the sensitive film. The sample gas collected with the flow rate of 5 ml/min and the

collection volume was varied from 5~25 ml. The column temperature was held at 50 °C for 2 min and then raised to 180 °C at 10 °C/min.

4. Result and discussion

Fig. 3 shows an example chromatogram of 10 ml collected by the dynamic HS method. **Fig. 4** shows the relationship between the peak areas of isoamyl acetate (peak 3), ethyl caproate (peak 5), and ethyl caprylate (peak 6), which are important aroma components of *sake*. In the static HS method, the peak area increased almost linearly with increasing collection volume. The peak areas of the dynamic HS method are close to that of the static HS method indicates that the aroma component in HS almost reached its equilibrium concentration in a short time. Although the peak area of the dynamic HS method tends to be smaller than that of the static HS method when the collected volume exceeds 20 ml, the monotonous increase with the collected volume is sufficient for quantitative analysis.

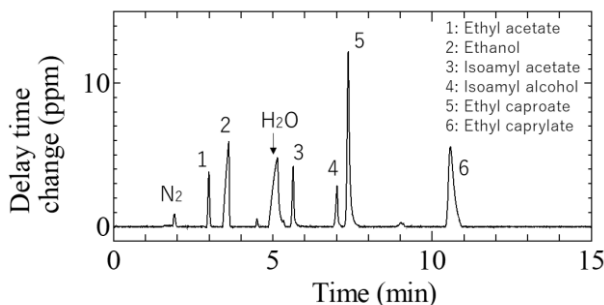


Fig. 3 Chromatogram of *sake* by ball SAW GC.

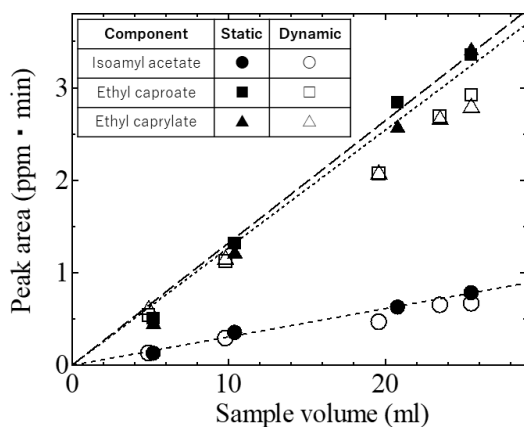


Fig. 4 Comparison of peak areas of main aroma components of *sake* by static HS analysis and dynamic HS analysis.

Fig. 5 shows the relationship between the peak areas of ethyl acetate (peak 1) and isoamyl alcohol (peak 4) and the collected volume. The static HS and dynamic HS methods showed comparable values, indicating that these components also reached almost

equilibrium concentrations of HS aroma components in a short time. On the other hand, the increase in peak area slowed down when the collection volume exceeded 10 ml. This may be due to the fact that the adsorbent in the concentrator exceeded the amount it could hold and passed through the concentrator without being adsorbed. Therefore, it was found that collection of less than 10 ml was effective for quantitative analysis of these components.

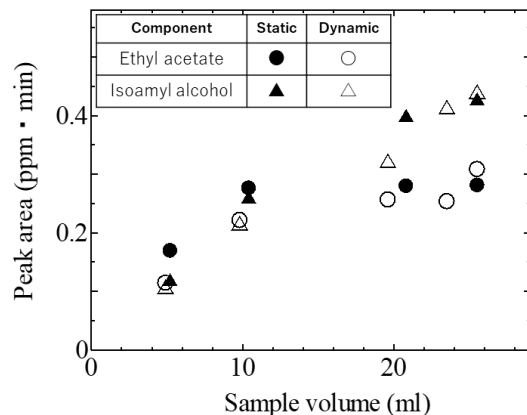


Fig. 5 Comparison of peak areas of ethyl acetate and isoamyl alcohol by static HS analysis and dynamic HS analysis.

5. Conclusion

In this study, it is shown that the dynamic HS method can collect a similar amount of aroma components to the static HS method and may be used for quantitative evaluation. Therefore, the dynamic HS method using the ball SAW GC can easily collect a large volume of sample gas for quantitative evaluation, and is expected to provide highly sensitive and accurate analysis of aroma components in food products in the field.

References

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